

## **APPENDIX C – LEAD AND ARSENIC SOIL CHARACTERIZATION AND TREATABILITY STUDIES**

### **C.1 INTRODUCTION**

Treatability studies were performed to assist in the selection of appropriate Cleanup Actions for impacted soils at the former DuPont Works Site (the "Site"). The studies were selected to address constituents impacting significant soil volumes; these constituents were lead and arsenic. This document will address: the requirements for treatability studies; the basis for treatability study selection and design; the treatability study approach and initiatives taken; a summary description of the treatability studies completed and the study results; a summary of interpretations made; and finally, conclusions drawn from the treatability studies and recommendations for additional treatability work.

### **C.2 TREATABILITY STUDY REQUIREMENTS**

The specific requirements of the treatability studies were determined based on the need to generate technical data to assist with the screening and selection of potential soil remediation technologies for lead- and arsenic-impacted soils. The lead and arsenic studies were designed with regard to the principal candidate technology categories identified through preliminary Feasibility Study (FS) screening. Chapter 4 provides an overview of the type of data required to assess the effectiveness of these types of technologies. The constituent-specific portions of Chapter 5 (5.3 and 5.4) contain a discussion of the treatability study results and their relevance to the evaluation of each technology category.

The treatability studies were carried out to meet the objectives described in Chapter 3. They include characterization studies performed by Hazen Research, Inc. (Hazen), in addition to speciation work, screening studies, and bench-scale technology evaluations performed by Hart Crowser, Battelle Pacific Northwest Laboratories, the State University of New York, the University of Cincinnati, and Weyerhaeuser Analytical and Testing Services.

### **C.3 BASIS FOR TREATABILITY STUDY SELECTION AND DESIGN**

#### **C.3.1 *Treatability Study Objectives***

The general objectives of the overall treatability study program, together with specific objectives with respect to soil characterization, are set out below.

- Provide sufficient information necessary to fully define the soil remediation problems presented and determine the specific requirements of the candidate technologies.
- Expedite the evaluation process prescribed by Model Toxics Control Act (MTCA) and so assist the selection of appropriate Cleanup Action Alternatives (CAA's) for soils impacted by the major constituents of concern, i.e.,
  - Provide information/data on treatment technology effectiveness;
  - Enable CAA's to be compared and evaluated on an equal basis;
  - Provide, in conjunction with available relevant literature, sufficient characterization and performance data to allow treatment alternatives to be developed and evaluated during FS detailed analysis;

- Help reduce performance uncertainties for treatment alternatives to acceptable levels to enable remedy selection; and
- Support the Interim Source Removal program.
- Provide preliminary supporting data for the design of selected CAA's and help define further data requirements needed prior to Cleanup Action implementation.

*Specific soil characterization objectives are:*

- To help fill gaps in physical and chemical Site characterization data pertinent to soil handling and to the treatment options developed;
- To determine, in detail, the physio-chemical nature of constituent/soil matrix interactions and thereby answer basic questions regarding constituent distribution and speciation, soil bonding/binding characteristics, and constituent leachability/mobility; and
- To provide information on the prospects for reducing volumes of significantly impacted soils through the use of physical screening techniques to segregate soils by particle size.

### **C.3.2 Rationale and Constraints**

The rationale for the selection and design of the treatability study program was based on several basic criteria. These criteria, which in some cases represent constraints on the scope of the program, are summarized below.

- The treatability studies should address the major constituents of concern under the FS, i.e., those impacting significant (greater than 5,000 yds<sup>3</sup>) soil volumes at levels exceeding the Working Hypothesis screening levels. These constituents of concern are lead and arsenic.
- The studies should be tailored to obtain appropriate data to assist in the determination of the technical viability of the principal candidate remediation technology categories identified through preliminary FS screening (see Chapter 2). The range of data obtained via the studies should reflect the broad range of innovative technology types and treatment approaches under consideration.
- The studies should be capable of fully characterizing the impacted soil matrices and the complex soil/chemical interactions (this criterion is further addressed in the opening discussion under Section C.4 below).
- The scope and duration of the studies should be consistent with the schedule for completion of the Remedial Investigation (RI) and FS activities.

## **C.4 TREATABILITY STUDY APPROACH AND INITIATIVES**

U.S. Environmental Protection Agency (EPA) analytical protocols are widely used in environmental remediation programs to determine the appropriate designation of potential 'waste' materials. These protocols include measuring the total or leachable concentrations of chemical constituents in the subject material (e.g., using SW-846 and Toxicity Characteristic Leaching Procedure (TCLP) methods), as well as testing the material for the characteristics of ignitability, corrosivity, reactivity, and toxicity. The protocols are designed to assist in determining whether any regulatory waste classification is applicable, so that the material may be managed, treated, and disposed of appropriately.

However, when dealing with a complex matrix such as soil impacted by chemical constituents, additional characterization information regarding physical and chemical properties and constituent/soil interactions is needed. In addition to assisting the assessment of regulatory classification, potential impact to human

health and the environment, and decisions regarding cleanup goals, this information is necessary to thoroughly evaluate which treatment technologies and processing conditions are applicable.

In this case, the treatability study approach principally involved a series of carefully devised laboratory tests directed at detailed characterization of representative soil samples impacted by the major chemical constituents of concern. The characterization testing program was designed with emphasis placed on the examination of complex soil/constituent (metal) interactions. The types of soil characterization information obtained relate to how constituents are held/bound in the soil matrix, what chemical form (speciation) constituents exist, and in what soil fractions (distribution) they reside. Associated information was also obtained to describe the soil composition (chemical and mineral content as well as particle size distribution) and assess the mobility/leachability of the bound constituents. The overall intent of the soil characterization studies was to provide a wide range of physical and chemical data giving insight into the viability and suitability of the principal candidate remediation technology categories referenced in Chapter 2, and to provide an efficient and cost-effective means of narrowing the field of potential Cleanup Action Alternatives. The physical analyses were primarily aimed at providing data, which will indicate how the soils will behave physically during materials handling, and physical treatment steps, while the chemical and speciation analyses were directed more towards identification of technological applicability.

The soil characterization methods and procedures described, by category, in the following paragraphs were selected for use in the lead and arsenic constituent initiatives, based on their ability to physically and chemically characterize metals-impacted soils. Further information is provided in Tables C-1 and C-2, "Soil Characterization Procedures", which lists each of the analyses included in the characterization studies (performed by Hazen), and specifies the type of information that can be determined from each analysis. The three categories presented below are used as the basis for the presentation and discussion of the treatability study results in Section C.5.

#### **C.4.1 Regulatory Analyses**

Regulatory analyses are designed to determine the regulatory classification of the waste, so that the waste may be managed, treated, and disposed of properly. Regulatory analyses include the RCRA Appendix IX list inorganics and semi-volatile organics. The Toxicity Characteristic Leaching Procedure (TCLP) was performed to determine whether the soil exhibited the RCRA characteristic of toxicity, and therefore should be assigned an EPA Characteristic Hazardous Waste Number.

#### **C.4.2 Physical Analyses**

Physical analyses are aimed at gathering data to indicate how the soil will behave physically during excavation, materials handling, and treatment processes. The physical analyses include particle size distribution and Atterberg limits (liquid limit, plastic limit, and plasticity index) that are used to determine the standard USDA soil classification. Other physical properties measured are moisture content and bulk density of both loose and packed soil.

#### **C.4.3 Chemical Analyses**

Chemical analyses are aimed at gathering information on soil chemistry, soil mineralogy, soil/constituent interactions, and constituent distribution, speciation, and mobility. The pH and Generalized Acid Neutralization Capacity (GANC) are measured of the soil's alkalinity or acidity and inherent buffering capacity. GANC and Reverse GANC data can also be used to assist the potential effectiveness of stabilization and soil washing/leaching treatments. The TCLP and Equilibrium Leaching Procedure (ELP) are both measures of leaching or mobility of the constituent. Organic carbon and anions can bond with heavy metals, reducing their mobility. Soluble cations can compete with heavy metals for bonding sites, increasing heavy metal solubility. X-ray diffraction, scanning electron microscopy, and sequential extractions are all used to determine which specific chemical form of the metal is present in the soil and how it is bound

The data quality objectives under the characterization studies were primarily to generate high quality technical data for use in performing evaluations and supporting positions developed under the FS. Only those procedures intended to determine the regulatory classification of the soil were performed strictly according to EPA SW-846 protocols. Under subcontract to Hazen, Evergreen Analytical, Inc. performed the regulatory analysis for the RCRA Appendix IX total inorganics (including lead, arsenic, and mercury) and semi-volatile organics, TCLP, and TCLP leachate organics and semi-volatile organics. The remainder of the analyses, performed by Hazen, was not completed according to strict SW-846 analytical protocols; however, every effort was made to ensure high quality data were generated. This approach was consistent with the main goal of this program, to generate high quality analytical information for technology screening purposes, rather than to produce legally defensible regulatory classification data. All procedures are documented and commercially available.

To supplement the laboratory characterization testing outlined above, additional speciation screening studies, and bench-scale treatability work was carried out to assist with a more detailed examination of the viability of soil washing and solidification/stabilization technologies.

## **C.5 SUMMARY DESCRIPTION AND RESULTS OF TREATABILITY STUDIES COMPLETED**

### **C.5.1 Introduction**

The majority of the treatability study work is represented by the laboratory and bench-scale soil characterization studies performed under contract by Hazen at their facility in Golden, Colorado. These studies involved soil samples from Site Areas known to be impacted by lead and arsenic, and include detailed characterization and speciation analysis of the lead- and arsenic-impacted soils. Soil sampling for the characterization studies utilized a statistical procedure based on EPA SW-846 guidance, recognized technical literature on sampling and analysis, and the experience of the Project Team. The sampling procedure enabled recovery of representative composite samples of impacted soils from randomly sampled grids in designated Areas of the Site determined, from pre-RI and RI information, to contain elevated concentrations of lead and arsenic.

The Hazen studies were supplemented by several additional specific initiatives performed by other commercial organizations and Universities, as outlined in Section C.5.2 below. Sections C.5.3 and C.5.6 present summaries of the constituent-specific work, focusing on the Hazen characterization studies, and include interpretations of the treatability study data generated. In the case of the lead-impacted soils characterization study (included in Section C.5.3), the work was part of a broader multi-site program conducted by DuPont to characterized company-wide lead remediation needs. The scope and detail of the DuPont study were greater than necessary for the purposes of this FS; only pertinent parts of the study and results are summarized here.

### **C.5.2 Other Initiatives Supporting Hazen Studies**

Hazen's work was supplemented with subsequent treatability study initiatives performed by other commercial organizations and Universities. These are listed below with a brief description of the study scope for each.

#### **BENCH-SCALE SOIL LEACHING STUDY – STATE UNIVERSITY OF NEW YORK, BUFFALO**

A screening evaluation of the non-proprietary soil leaching chemistries was performed by the Department of Civil Engineering at the State University of New York at Buffalo (UB). The efficacy of various soil washing extraction agents and leaching conditions were evaluated for removing lead from a lead-impacted soil sample (-8 to +200 mesh fractions) from Area 40 (Pulverizer) of the Site, provided by Hazen. The results of this soil leaching study are contained in the final report prepared by UB researchers entitled Evaluation of Soil Washing Extraction Agents and Processes for Remediating Heavy Metal Contaminated Soil from Weyerhaeuser Area 40 Pulverizer," March, 1994.

### **SOLIDIFICATION/STABILIZATION SCREENING STUDY – UNIVERSITY OF CINCINNATI**

The University of Cincinnati Center for GeoEnvironmental Science and Technology (CGEST) conducted solidification/stabilization screening tests on soil fines (-200 mesh) samples from Areas 36 and 40 (Glazemill and Pulverizer) of the Site provided by Hazen to evaluate the ability of three non-proprietary binder mixes to solidify and stabilize lead-impacted soils. The effects of lead-designed solidification/stabilization binder mixes on arsenic contained in the Area 36 sample were also investigated.

The results of this screening study are contained in the final report prepared by CGEST researchers entitled "Solidification/Stabilization Screening for Integrated Lead Remediation Program", 1993.

### **BENCH-SCALE SOIL LEACHING STUDY – WEYERHAEUSER ANALYTICAL & TESTING SERVICES**

To investigate the potential for utilizing non-proprietary leaching chemistries to remove arsenic from the Site soils, a leaching study was carried out at the laboratories of Weyerhaeuser Analytical and Testing Services (Weyerhaeuser ATS). Under this three-phase study, several leaching solutions were applied to - 1/4" by +200 mesh fractions of combined soil samples from Area 18 and the Narrow Gage Railroad area. The effects of pH and liquid:solid ratio were investigated as well as performance under single-stage batch and simulated multistage countercurrent extraction regimes.

### **ARSENIC SPECIATION ANALYSIS – BATTELLE PACIFIC NORTHWEST LABORATORY**

Battelle Pacific Northwest Laboratory (Battelle PNL) performed limited arsenic speciation analysis on soil samples from Areas 5, 25, 38, and the Narrow Gage Railroad area. The purpose of this study was to identify ionic species of arsenic and investigate the presence of organic arsenic forms in Site soils potentially impacted by acid production (Areas 5 and 25), the use of arsenic-based herbicides (Narrow Gage), or arsenic-based paint/ink (Area 38).

### **WET SCREENING TESTING ON ARSENIC-IMPACTED SOIL – HART CROWSER**

Additional arsenic treatability studies were performed by Hart Crowser to better define the distribution of arsenic concentration with respect to soil particle size, through the use of particle size distribution testing and associated arsenic analysis of sized soil fractions. Samples were taken in the 250/500 – foot arsenic soil sampling grid area (including Areas 18 and 36) and the Narrow Gage Railroad area to supplement the results from the Hazen arsenic characterization study completed on samples from the same areas.

#### **C.5.3 Lead-Impacted Soils**

Fifty-five gallon composite soil samples were collected from each of the following Consent Decree Areas, one sample per Area: Area 7 (Kettle), Area 18 (Nitrator Number 1), and Area 36 (Lead Melting). Three soil samples were also collected from Area 40, one each from the Pulverizer, Glazemill, and Packhouse locations. The method of sample collection was detailed in the sampling plan for each Area; a brief summary is described below.

Previous sampling results were used as a basis for establishing sampling grids in each Area. For each grid area (containing 100 nodes), a random number generator was used to select 25 discrete sample locations. Sub-areas, within the identified Consent Decree Areas, shown by previous sampling results to exhibit lead concentration below the MTCA Method A screening level of 250 mg/kg were not included in the sampling regime. Only soils that contained lead concentrations greater than 250 mg/kg were included (i.e., "hot spots" were in effect, sampled).

The soil samples for each grid area were taken from shallow depth intervals that were chosen to represent the soil to be potentially remediated based on previous vertical delineation. Approximately two gallons of soil were collected from each of the 25 selected sampling locations. The 25 samples were then composited into one 50-gallon samples from each area samples were shipped to Hazen in 55-gallon drums.

Composite sampling was chosen to adequately represent each area because the resulting soil homogenization is a simulation of the mixing that would occur during full-scale excavation and ex-situ

treatment. It is not significant that this sampling approach did not allow a thorough evaluation of lead concentration variability on technology performance, since these issues will be addressed in any future pilot testing.

Upon receipt of the soil samples, Hazen personnel prepared and tested the samples as outlined in Figure C-1, "Flow sheet for Sample Preparation and Testing". Portions of the samples from Areas 36 and 40 (Glazemill and Pulverizer) were sent to CGEST for the solidification/stabilization screening study, and portions of the sample from Area 40 (Pulverizer) were sent to UB for the bench-scale soil washing evaluation.

#### **C.5.4 Test Results**

Complete results for all of the procedures conducted under the lead characterization study are detailed in the final reports prepared by Hazen for each of the six samples tested. The reports are entitled "Laboratory and Bench-Scale Studies for Characterizing Lead-Contaminated Soil from DuPont/Weyerhaeuser Remediation Site", (August-October, 1993). The characterization results are summarized in Table C-3, "Summary of Lead-Soil Characterization Test Results"; the results are presented according to the information categories indicated in Section 4.0. An expanded summary of selected results is presented below in the same format as Table C-3, with limited interpretation.

#### **REGULATORY ANALYSIS**

The total lead concentrations, as determined by Method 6010 as part of the RCRA Appendix IX metals analysis, ranged from 260 mg/kg to 3,000 mg/kg for the six composite soil samples tested. The TCLP lead analyses results indicate that the soil samples from Areas 7, 18, 36, and 40 (Pulverizer and Glazemill) were not RCRA D008 hazardous, based on the regulatory limit of 5 mg/l. However, the soil sample from the Packhouse area of Area 40 exceeded the RCRA D008 regulatory threshold with a TCLP lead result of 18 mg/l; the corresponding total lead assay for this sample was 3,000 mg/kg.

TCLP analysis for the other metals and organics was also conducted. The results indicate that the Site soil does not exhibit the RCRA toxicity characteristic for any of these constituents.

There were other RCRA Appendix IX metals present, but these were at very low concentrations in all cases (the highest values were in the Packhouse and Pulverizer areas of Area 40, with zinc concentrations up to 2,500 mg/kg and moderate concentrations of copper and barium). No RCRA Appendix IX semi-volatile organics were present in the soil samples from Areas 7 and 40 (Glazemill and Packhouse). Extremely low levels (ppb) of semi-volatiles were present in the samples from Areas 18, 36, and 40 (Pulverizer). The semi-volatile constituents were polynuclear aromatic hydrocarbons (PAHs), which appear to be related to petroleum-based materials previously identified at the Site.

#### **PHYSICAL ANALYSIS**

Physical testing determined that the USDA standard soil classification was "sand" for the samples from Areas 7, 36, and 40, and "sandy loam" for the sample from Area 18. The results of the Atterberg Limits tests indicate that all six-soil samples were non-plastic, suggesting that soil excavation and processing can be managed in a straightforward manner.

The particle size distribution test results for the soil samples ranged from 38 to 61% cobbles/gravel (greater than 4 mesh), 32 to 53% sands (less than 4 and greater than 200 mesh), and 7 to 15% silt/clay (less than 200 mesh). This data indicates that the soil is amendable to standard physical screening or size classification-type soil washing treatment.

The moisture content results ranged from approximately 5 to 20%; loose and packed densities ranged from 1.1 to 1.6 grams/cubic centimeter (g/cm<sup>3</sup>) (or approximately 0.9 to 1.3 tons/cubic yard (tons/yd<sup>3</sup>)) and from 1.4 to 1.9 g/cm<sup>3</sup> (or approximately 1.2 to 1.6 tons/yd<sup>3</sup>), respectively. The density data is given to represent ex-situ and in-situ soils, respectively, and reflects typical ambient moisture.

The pH of the soil samples ranged from 4.4 to 7.4 in a slurry (10% solids) of soil and demineralized water. Since the soil is mildly acidic to neutral, it will not be corrosive to excavation, materials handling, and other treatment equipment.

The physical analyses data collectively indicate that materials handling of the Site soils, including provisions for dust control should be uncomplicated and accomplished using conventional equipment and methods.

### **CHEMICAL ANALYSIS**

Lead assays performed using Method 7420 on pulverized heads of whole soils for each of the six samples determined total lead concentrations (average of three readings) in the range of 180 to 3,600 mg/kg.

Anions considered likely to produce metal salts of low solubility were investigated in the soil samples. Those found to be present were sulfide, sulfate, phosphate, and carbonates. Total concentrations of these low solubility bonding/binding anions present in the samples from Areas 7, 18, and 36 ranged from approximately 2,500 to 2,650 ppm. The total concentrations of these anions in the samples from Area 40 ranged from approximately 4,200 to 10,700 ppm. In general, the level of anions in the soils is substantially greater (up to 10X) than the total lead concentration, indicating that low solubility metal salts can potentially form. Evidence of such compounds in the soils was provided using more sophisticated speciation techniques, as discussed in later sections.

The presence of soluble cations, which can compete with any soluble lead species for soil adsorption sites, soil ion exchange sites and binding anions, was also screened. Competing cations investigated were calcium, sodium, magnesium and potassium. The total leachable concentration of these cations present in the soil sample from Area 18 was very low at less than 50 ppm. The total leachable concentrations of cations present in the samples from Areas 7, 36, and 40 (Glazemill and Packhouse) were low, ranging from 50 to 100 ppm. The total leachable concentration of cations present in the soil sample from the Pulverizer area of Area 40 was also low at less than 300 ppm. These results indicate that soluble lead should be able to bind with soil at adsorption and ion exchange sites since competition is not severe. The results are consistent with sequential extraction speciation results discussed later.

Organic carbon (organic matter) content is important as it can bind with heavy metals and can potentially interfere with soil treatment processes. The organic carbon present in the soil samples from Areas 7, 18, and 36 ranged from 0.9 to 3.3%; no detectable levels of inorganic (fixed) carbon were reported for these samples. The organic carbon present in the samples from Area 40 ranged from 4.2 to 6.9%; the inorganic carbon content of these samples ranged from non-detect to 0.7%. These levels are considered moderately low from a soil treatment perspective and should not interfere with implementation or performance of soil washing, solidification/stabilization and other technologies. Sequential extraction was used to determine how much lead was associated with organic matter in the soil.

The results of X-Ray Fluorescence analysis (XRF) indicate the presence of a soil matrix comprising silica, alumina and iron oxide, with minor amounts of sodium, magnesium, potassium, phosphorus, sulfur, titanium and calcium. These results are to be expected for the type of soils present at the Site.

The Generalized Acid Neutralization Capacity (GANC), Reverse GANC (RGANC), and pH analyses data indicate that the soil samples ranged from slightly acidic to neutral and had little acidic or basic buffering capacity.

Results of the Sequential Extraction tests performed on selected sized fractions indicate that the majority of the lead was present in carbonate, specifically adsorbed, and in ion-exchangeable forms in samples from Areas 7, 18, and 36. For the Area 40 samples, the majority of the lead was shown to be associated with carbonates, iron and manganese oxides, and organic material.

Speciation analyses performed on selected sized fractions (gravity separated as appropriate) using Scanning Electron Microscopy (SEM) and X-Ray Diffraction (XRD) gave positive identification of lead in the following forms: lead carbonate, lead oxide, lead-iron oxide, lead-manganese oxide, lead-tin oxide, lead-aluminum oxide, lead sulfate, lead sulfide, lead silicates, lead phosphates, and metallic lead.

### **LEACHING/MOBILITY**

The TCLCP lead analytical results are summarized in the Regulatory Analysis section, above.

The Equilibrium Leaching Procedure (ELP) results demonstrate very low lead solubility with synthetic acid rain (0.02 to 0.06 mg/l). The wet screening and Sequential Extraction test results also demonstrate that the lead species are relatively insoluble in water. However, the lead was more soluble in acetic acid solutions, as demonstrated by TCLP, GANC, and Sequential Extraction test data. These results indicate that lead is essentially immobile under normal environmental conditions, but can be mobilized under more rigorous leaching conditions.

Under the GANC and RGANC tests, one stage acidic and alkaline leaching at 20:1 liquid/solids ratio removed a range of 45 to 75% of the lead using 2N (normal) acetic acid, and a range of 13 to 40% of the lead using 2N sodium hydroxide. This indicates that lead is more readily mobilized under acidic, as opposed to basic, leaching conditions, but also indicates the presence of some extremely leach-resistant forms of lead.

### **SOLIDIFICATION/STABILIZATION SCREENING – UNIVERSITY OF CINCINNATI (CGEST)**

Three non-proprietary binding mixes (two containing Portland cement and one containing a combination of cement kiln dust and fly ash) were used to screen solidification/stabilization technology, using -200 mesh fractions of soil samples from Areas 36 and 40 (Glazemill and Pulverizer) supplied to CGEST by Hazen. The following are the main results after 28 days of curing:

- All three design mixes passed TCLP, with lead leachate results ranging from 0.29 to 1.5 mg/l;
- The amount of lead that leached in the TCLP tests ranged from 0.44 to 2.73% of the total lead in the treated sample (initial untreated soil lead concentrations ranged from 2,900 to 3,200 mg/kg);
- The end point pH in the TCLP tests ranged from 5.2 to 8.2; and
- Unconfined Compressive Strength (UCS) data for the prepared samples of fine material with Portland cement binder ranged between 18 and 96 pounds per square inch. The fine material treated with kiln dust/fly ash did not set within the 28-day curing time and strength analysis could not be completed.

### **BENCH-SCALE SOIL WASHING STUDY – STATE UNIVERSITY OF NEW YORK, BUFFALO (UB)**

The Department of Civil Engineering at the UB examined soil leaching through laboratory and bench-scale experiments. The efficacy of various soil leaching extraction agents and conditions was evaluated for removing lead from a lead-impacted soil sample (-8 to +200 mesh fractions) from Area 40 (Pulverizer). The results relevant to the evaluation of treatment technologies are summarized below:

- Soil lead was reduced to less than one-half the initial mean concentration of 1,401 +/- 244 mg/kg (95% confidence interval) in approximately one-third of the soil leaching experiments conducted.
- The lowest soil lead concentration (444 +/- 24 mg/kg) was achieved using hydrochloric acid at a pH of 1 with a liquids/solids ratio of 10:1, and at 25 deg C;
- Strong mineral acid extractants containing the chloride ion (HCl, HClO<sub>4</sub>) were the most effective at removing lead from the soil;



- Significant lead removal (>50%) was achieved with an HC1 extraction solution at more moderate pH (~3) with the addition of EDTA as a complexing (chelating) agent; and
- TCLP lead values for the treated soil samples ranged from 0.3 to 0.5 mg/l.

### **C.5.5 Interpretation of Results**

The Hazen soil characterization results which bear most significantly on the viability and effectiveness of solidification/stabilization, physical screening/soil washing, and smelting/thermal recovery technologies for remediating lead-impacted soils are listed in Table C-3. A brief discussion of key results from the characterization study, including implications for technology selection, is provided below. Results of the UB bench-scale soil washing tests and the CGEST solidification/stabilization screening tests are also discussed briefly, as appropriate.

#### **SOLIDIFICATION/STABILIZATION**

As indicated in Table 3, the characterization test information most relevant for the evaluation of solidification/stabilization remediation technology are GANC/RGANC, Metals Acid Neutralization Capacity (MANC)/Reverse MANC, organic carbon, RCRA Appendix IX semi-volatiles, TCLP, and moisture content.

As previously mentioned, the GTANC, RGANC, and pH analyses indicate that the Site soils sampled are mildly acidic to neutral and have little acidic or basic buffering capacity. The low buffering capacity is advantageous for a solidification/stabilization process because a minimum amount of binder would need to be added to buffer the soil, add reserve alkalinity, and drive lead solubility to low values. Based on the GASNC and MANC plots, the optimal pH (corresponding with the minimum lead solubility) for the Site soils for solidification/stabilization treatment lies in the range of moderately acidic to moderately basic (pH 4-10).

The carbon analysis results indicate that the organic carbon content in the Site soils (0.9 to 6.9%) is typically below levels (<10%) that would be expected to inhibit curing in a solidification/stabilization process. However, with regard to the stabilization of soil fines (-200 mesh), the CGEST study indicates that fly ash/kiln dust binders require longer curing times than Portland cement binders.

The RCRA Appendix IX semi-volatiles results indicate that semi-volatiles are either absent in the Site soils or present only in very low concentrations. These results indicate the application of a straightforward solidification/stabilization treatment. Some semi-volatile organics (sugars, alcohols) can be difficult to solidify/stabilize and may inhibit curing of treated material.

Only one of the six samples analyzed for TCLP exceeded the RCRA D008 regulatory threshold of 5 mg/l, the Area 40 (Packhouse) soil sample (18 mg/l). No other constituents exceeded TCLP threshold levels. These results indicate that if solidification/stabilization treatment were applied to the Site soils, the TCLP lead leachability criterion would be readily met. The solidification/stabilization screening performed by CGEST further supports this conclusion. Here, several conventional binding mixes were tested on soil fines from Areas 36 and 40 (Glazemill and Pulverizer) and yielded TCLP results well below the 5 mg/l RCRA Land Disposal Restriction (LDR) treatment standard, on the stabilized soils (0.29 to 1.5 mg/l).

The moisture content results for the Site soils were variable, ranging from approximately 5 to 20%. Moisture content is the most sensitive input variable in solidification/stabilization system design and requires careful control during implementation. At the levels measured in this case, based on the CGEST solidification/stabilization screening work where moisture contents were in the range of 40 to 50%, a requirement for make-up water is indicated.

#### **PHYSICAL SCREENING/SOIL WASHING**

The characterization test results of most relevance for the evaluation of physical screening/soil washing remediation technologies (as shown in Table C-3) are silt-clay content, lead distribution with regard to

particle size, surface area calculations, Atterberg Limits, organic carbon, GANC/RGANC, MANC/RMANC, and Sequential Extraction.

The silt/clay content of the Site soils, ranging between 7% and 15% for the samples tested, is quite low, and well below the 40% upper limit generally considered likely to limit the effectiveness of soil washing processes. The lead content in the silt/clay fraction of the Site soils is substantial at between 27% and 67% (corresponding concentrations range from 2,170 mg/kg to 10,600 mg/kg). These results suggest that significant beneficial volume reduction of lead-impacted soil could be achieved by separating the fine material from the whole soil.

Wet screen particle size distribution test results and corresponding chemical analyses results for separated size fractions indicate that between 84% and 97% of the soil lead is concentrated in the sand and silt/clay fractions. These fractions represent between 40% and 62% by weight of the whole soil. These results indicate that recovery of the cobbles/gravel fraction (representing between 38% and 60% of the whole soil) as “clean” material could be readily achieved using conventional wet screening or classification-type soil washing.

For water-based soil washing processes, the wet screening and Sequential Extraction test results indicate that, due to the low aqueous solubility of lead in Site soils, little or no water treatment provisions would be necessary.

The surface area calculations for each of the soil size fractions indicate, as expected, that the majority of the total soil surface area is contained in the silt/clay (64% to 83%), with less in the sands (17 to 36%), and least in the cobbles/gravel (<1% to 2%). In the case of Areas 7 and 18, the lead distribution by size fraction increases with reducing size fraction and increasing surface area; these results indicate that lead bonding and binding is mostly surface related. The lead distribution for Areas 36 and 40 in the sand and silt/clay fractions does not proportionally follow surface area distribution; this indicates that lead is not only surface bound but is also associated with other particulate matter present in the soil matrix. In the case of Area 36 soils, SEM confirmed the presence of particulate lead.

The Atterberg Limits results show that the “sand” and “sandy loam” soil is non-plastic and should be both amenable to processing by physical screening equipment and easy to handle with conventional earth-moving equipment.

The organic carbon content of the Site soils (0.6% to 6.9%) is below the 10% limit generally considered as the value above which complications in a soil washing system can arise due to contaminant-carbon bonding. The inclusion of an organic carbon removal unit operation should not be necessary.

The GANC/RGANC results, as mentioned previously, show that the Site soils have little buffering capacity and would not require the addition of large amounts of acid in a chemically enhanced soil-washing scenario, to achieve a desired pH change.

The MANC/RMANC equilibrium lead concentrations in the aqueous phase occur between additions of 2 to 40 equivalents of acid/kg soil and between additions of 2 to 40 equivalents of base/kg. One stage acidic and alkaline leaching at 20:1 liquids/solids ratio can remove 45% to 75% of the lead using 2N acetic acid, or 13% to 40% lead using 2N sodium hydroxide. This indicates that acidic leaching has potential for use in a soil washing system to clean the Site soils, possibly in conjunction with wet screening and other physical (gravity, density, and high-energy attrition) treatment techniques. It is important to note, however, that even under severe leaching, lead removal is not complete as can be explained by the presence of some extremely leach-resistant forms of lead.

Sequential Extraction data show that lead is predominantly present, in the sized fractions examined, as carbonate, specifically adsorbed, and non-exchangeable forms, as well as being associated with iron and manganese oxides and organic material. This speciation information, which is supported by the SEM and

XRD results, also indicates that further lead removal could be achieved in a chemically enhanced (acidic) soil washing or leaching treatment scenario.

The results of the UB soil washing study provide further support for considering a chemically enhanced soil washing or leaching treatment application. The study showed that strong mineral acid extractants (specifically, hydrochloric and perchloric acid) at approximately pH 1 were effective at significantly reducing (by greater than 60%) lead concentrations of approximately 1,400 mg/kg in the sand fraction of Site soils. TCLP lead values for treated samples ranged from 0.3 to 0.5 mg/l. Less acidic conditions (pH 3) were also effective if a chelating agent (such as EDTA) was added. Other conclusions drawn from this study, such as those relating to the detailed behavior of complexing compounds and the effects of temperature and liquid/solids ratio, as well as aqueous extract treatment/recycling, will be essential issues to consider when deciding on whether to include leaching in a soil washing treatment approach.

### **SMELTING/THERMAL RECOVERY**

The characterization test results of primary importance for the evaluation of smelting/thermal recovery remediation technology are total lead concentrations, moisture content, RCRA F039 listed metals, and soil mineralogy results.

Cost effective treatment of RCRA D008 wastes by smelters requires that lead content in the feed soils be on the order of 6 to 8% by weight or that the soil contain at least 80% silica (silica is a flux agent used in smelting). Since total average lead concentrations in the Site soils lie in a range of only 180 to 3,600 mg/kg (i.e. 0.4% maximum), and even the highest measured concentration in the silt/clay fraction is only 1.1%, the soils would not be suitable for smelting without intensive pre-processing to concentrate lead into the finer soil fractions. Silica content for Site soils ranges from 30 to 60%, which also indicates that the soil is not suitable as a silica flux replacement in smelting. Though the possibility of using smelting to treat concentrated soil-washing residuals does still exist, the technical and economic viability would require further investigation.

The maximum acceptable moisture content of a smelter feed soil is 10%. This may place a further constraint on the smelting application for the Site soils; concentrated fines/residuals from a soil washing process would likely need to be passed through an advanced dewatering step.

The RCRA F039 metals analyses indicate the presence of other metals including: antimony, arsenic, barium, chromium, copper, nickel, selenium, vanadium, and zinc. Though present at low concentrations, these metals are potential impurities in certain smelter products that may restrict the facilities that could potentially handle the soils or treatment residuals.

The XRF analysis results indicate the presence of a silica-alumina-iron oxide matrix, with minor amounts of sodium, magnesium, potassium, phosphorus, sulfur, titanium, and calcium. The presence of these elements is expected in sandy and silty soils. They could, however, adversely affect the viability of smelting/thermal recovery as impurities in the system.

#### **C.5.6 Arsenic-Impacted Soils**

Soil samples for the Hazen study were collected from each of the following areas of the Site (one sample per area): Area 18, Area 36, and the Narrow Gage Railroad area. In addition to the characterization work performed by Hazen, Hart Crowser performed a laboratory wet screening study to better define the concentration distribution in relation to soil particle size on samples from these same areas. An additional five soil samples from the 250/500-foot arsenic sampling grid area (including Areas 18 and 36), and five samples from the Narrow Gage Railroad area were collected for the Hart Crowser study. The sample collection methods are summarized in the following paragraphs.

Previous sampling results were used as a basis for establishing sampling grids in each area. For the Area 18 and 36 grids, a random number generator was used to select 25 discrete sample locations. Some parts of the Site, within targeted areas, shown by previous sampling results to exhibit arsenic

concentrations below the Site background level of 32 mg/kg were not included in the sampling regime. The Narrow Gage area was segregated into 100 linear segments along the railroad track; 25 segments were randomly selected for collection of three-point composite samples, which were composited into a 55-gallon sample. For the Hart Crowser screening study, ten five-point composite samples were collected at locations identified during the RI (including the December 1993, Phase I Arsenic Sampling) as areas of elevated arsenic.

The soil samples for each grid area were taken from a shallow depth interval (0-1 foot) chosen to represent the soil to be potentially remediated based on previous vertical delineation. Composite sampling was chosen to adequately represent each area because the resulting soil homogenization is a simulation of the mixing that would occur under full-scale excavation and ex-situ treatment operations. Arsenic concentration variability issues will be more specifically addressed in any future pilot testing.

Approximately two gallons of soil were collected from each of the 25 selected sampling locations for the Hazen study. The 25 samples were then composited into one representative 50-gallon sample for each area and shipped to Hazen in 55-gallon drums. Upon receipt of the soil samples, Hazen personnel prepared and tested the samples. Portions of the samples from Area 18 and the Narrow Gage Railroad area were sent to Weyerhaeuser ATS for the bench-scale soil leaching study.

### **C.5.7 Test Results**

Complete results for all the procedures conducted under the arsenic characterization study are detailed in the final report prepared by Hazen for each of the three samples tested. The reports are entitled "Laboratory and Bench-Scale Characterization Studies for Arsenic-Contaminated Soil from DuPont/Weyerhaeuser Remediation Site," (March-April, 1994). The characterization results are summarized in Table 4, "Summary of Arsenic-Soil Characterization Test Results". The results are presented according to the information categories indicated in Section 4.0. An expanded summary of selected results is presented in the following sections, with limited interpretation. Further data interpretation, as applicable to specific remediation technology categories, is provided in Section 5.4.2.

### **REGULATORY ANALYSIS**

The TCLP arsenic analyses results for each of the soil samples were less than 0.07 mg/l and indicate that the samples were not RCRA D004 hazardous, based on the regulatory limit of 5 mg/l. TCLP analysis for the other metals and organics was also conducted on the sample from Area 36; the results indicate that the soil does not exhibit the RCRA hazardous toxicity characteristic for any of these constituents.

### **PHYSICAL ANALYSIS**

Physical testing determined that the USDA standard soil classification was "sand" for the soil samples from each of the three Areas. The results of the Atterberg Limits tests indicate that the samples from Areas 36 and 18 (by inference from analysis performed on a different composite sample from Area 18) were non-plastic.

The particle size distribution test results for the soil samples ranged from 52% to 66% cobbles/gravel, 26 to 39% sands, and 8 to 10% silt/clay.

For the Area 36 sample, the moisture content measured 5% and the loose and packed densities measured 1.6 and 1.9 g/cm<sup>3</sup> (or approximately 1.3 and 1.6 tons/yd<sup>3</sup>), respectively. Also, the pH of a soil slurry measured 6.3, which indicates that the soil will not be corrosive to excavation, materials handling, or other treatment equipment.

Many of the physical analyses performed on the lead samples were not considered necessary when studying arsenic-impacted soils because much of the general information gathered from the lead characterization/treatability studies could be applied to soils across the Site for the purposes of evaluating potential treatment technologies. The physical analyses data collectively indicate that materials handling of the Site soils will not be complicated.

## **CHEMICAL ANALYSIS**

Arsenic assays performed using Method 7060 on pulverized heads of whole soils for each of the three samples tested determined total arsenic concentrations in the range of 29 to 353 mg/kg.

Concentrations of low solubility bonding/binding anions and soluble cations, and organic carbon content for the Area 36 sample were measured at 2,640 ppm, 77 ppm, and 0.9%, respectively as reported under the lead characterization study. Although these analyses were not performed on the Area 18 sample, existing data from the lead characterization study composite sample from Area 18 indicates that the soils from Areas 18 and 36 have similar chemical characteristics.

The XRF analyses data indicate the presence of a silica=alumina-iron matrix with lesser amounts of sodium, magnesium, potassium, and calcium. These results are to be expected for the type of soils present at the Site and are consistent with the results from the lead characterization study.

The GANC/RGANC and pH analyses data show that the soil samples were essentially neutral and had no appreciable acidic or basic buffering capacity.

Sequential Extraction test results for selected sized fractions indicate that, for all samples, the occurrence of arsenic in the selected sized fractions examined was mostly in association with iron oxides and the organic portion of the soils.

Speciation analyses performed on selected sized fractions (gravity separated as appropriate) using SEM confirmed and Sequential Extraction test results that arsenic as associated with iron oxides and with the organic portion of the soil. SEM also gave positive identification of arsenic in the following forms or associations: iron-arsenic-phosphate, potassium-iron-sulfate, silicates, manganese oxides, and lead-arsenic-sulfate. An overall assessment of the SEM results suggests that arsenic exists predominantly in the +5 valence state.

## **LEACHING/MOBILITY**

The TCLP arsenic analytical results are summarized in the Regulatory Analysis section, above.

Under the GANC and RGANC tests, one stage acidic/basic leaching at 20:1 liquids/solids ratio removed a range of two to four percent of the arsenic using 2N (normal) acetic acid, and 74% to 80% using 2N sodium hydroxide, respectively. This indicates that arsenic is more readily mobilized under basic, rather than acidic, leaching conditions. In general, the solubility of arsenic in the RGANC test was ten times higher than the corresponding solubility in the GANC test. This can be explained by reference to the sequential extraction results, which indicate the predominant arsenic forms and associations identified in the Site soils. Arsenic compounds in the +5 valence state are soluble under oxidizing conditions and at high pH.

The arsenic was more soluble in weak sodium hydroxide solutions than under moderate acetic acid leaching conditions, as demonstrated by TCLP, GANC, and Sequential Extraction test data. These results indicate that arsenic is essentially insoluble and immobile under normal environmental conditions, but can be mobilized under more rigorous high pH (caustic) leaching conditions.

## **BENCH-SCALE SOIL LEACHING STUDY – WEYERHAEUSER ATS**

The potential of solutions of sodium hydroxide and sodium carbonate (at varying strengths, with and without phosphate addition) to extract arsenic from -1/4" to +200m fractions of the Site soil were investigated in single-stage batch extraction experiments under a study performed by Weyerhaeuser ATS. The effect of changes in liquid/solids ratio was evaluated, and the relative leaching efficiencies achieved for several sub-fractions of the soil sample examined. A simulation of a potential multi-stage countercurrent field extraction process was also conducted. The principal finds of this study are as follows:

- Soil arsenic concentrations were reduced by a maximum of 57% from a starting concentration of between 103 and 113 mg/kg in a single-stage batch extraction at an end point pH of 11.5 with 0.02 N sodium hydroxide and a liquid/solids ratio of 20:1. Similar performance was achieved with a sodium carbonate solution, although a significantly higher reagent dose (e.g. 0.33 N) was necessary.
- A phosphate solution at near neutral pH was able to reduce arsenic concentrations by up to 10%. Extraction efficiencies of up to 25% were achieved in experiments evaluating the performance of phosphate in conjunction with alkali; appreciable pH buffering by phosphate was, however, seen, particularly in solutions including sodium carbonate.
- Leaching performance was shown to vary as a function of end point pH under the extraction experiments (increased sharply with increasing pH over the range of reagent strengths tested).
- Caustic leaching performance was not constrained by arsenic solubility under tests involving liquid/solids ratios of between 5:1 and 20:1.
- A somewhat greater percentage reduction in soil arsenic concentration was seen for the larger sub-fraction in a single-stage batch extraction examining relative leaching efficiencies for three sub-fractions of the tested soil.
- The overall leaching performances achieved under single stage batch and simulated multi-stage countercurrent extractions were comparable.

#### **WET SCREENING TESTING – HART CROWSER**

A total of five composite soil samples from the 250/500-foot arsenic sampling grid area and five samples from the Narrow Gage Railroad area were taken for wet screening analysis. The analysis involved separation of the whole soil into a series of size fractions for subsequent total arsenic analysis. The data generated were combined with the wet screening data derived for arsenic-impacted soil samples from Area 18, Area 36 and the Narrow Gage Railroad area under the Hazen study, the presence of higher arsenic concentrations in the smaller size fractions was consistently shown by these results. The combined results were subjected to a statistical evaluation (involving calculation of cumulative mean arsenic concentrations with reducing size fraction) to allow various “cut points” to be examined in consideration of potential remediation scenarios featuring soil size separation.

#### **SPECIATION ANALYSIS – BATTELLE PACIFIC**

Data from the Battelle Pacific speciation study indicate that, in the areas of the Site sampled (Areas 5, 25, 38, and Narrow Gage Railroad), arsenic is largely present as As(V). As(III), the common reduced form, accounts for less than one percent of the total soil arsenic. One sample in the Narrow Gage Railroad area contained about four percent organic (methylated) arsenic, which indicates the possible use of organic arsenic-based herbicides on the Site.

#### **SOLIDIFICATION/STABILIZATION SCREENING – UNIVERSITY OF CINCINNATI (CGEST)**

Three non-proprietary binding mixes (two featuring Portland cement and one featuring a combination of cement kiln dust and fly ash) were used to stabilize the -200-mesh fraction of a soil sample from Area 36 supplied to the University by Hazen. The initial arsenic concentration in the soil fraction was 205 mg/kg. All three design mixes passed TCLP following a 28-day curing period, with arsenic leachate results below the method detection limit (<0.1 mg/l).

#### **C.5.8 Interpretation Results**

The soil characterization results which bear most significantly on the viability and effectiveness of solidification/stabilization, physical screening/soil washing, and smelting/thermal recovery technologies for remediating arsenic-impacted soils are listed in Table C-2. A brief discussion of key results from the

characterization study, including implications for technology selection, is provided in the following sections.

The results of the CGEST solidification/stabilization screening tests, the Hart Crowser wet screening testing, and the Weyerhaeuser ATS bench-scale soil leaching study are also discussed briefly, where appropriate.

### **SOLIDIFICATION/STABILIZATION**

The characterization test information most relevant for the evaluation of solidification/stabilization remediation technology are GANC/RGANC, organic carbon, RCRA Appendix IX semi-volatiles, TCLP (inorganic and semi-volatiles), and moisture content.

As previously mentioned, the GANC/RGANC and pH analyses indicate that the Site soils sampled are neutral and have essentially no acid or basic buffering capacity. The low buffering capacity is advantageous for a solidification/stabilization process because it minimizes the amount of solidification/stabilization binder that would need to be added to buffer the soil and maintain the arsenic solubility at low values. Based on the GANC and RGANC plots only, the optimal pH for the Site soils (corresponding to minimum solubility) for solidification/stabilization treatment of arsenic is in the acidic range (pH 3 to 4). This is consistent with the speciation results since As(V) species are known to be very insoluble in acid. To ensure a practical treatment and sensible pH conditions, the target end point pH under TCLP testing should be only a slightly acidic pH 5 to 7.

The carbon analysis results indicate that the organic carbon content in the Area 36 Site soils (<1%), which is a measure of the soil's natural ability to bond and bind with arsenic and other constituents, is typically below levels (<10%) that would be expected to inhibit curing in a solidification/stabilization process. With regard to the stabilization of soil fines (-200 mesh), the CGEST study indicates that fly ash/kiln dust binders require longer curing times than Portland cement binders.

The RCRA Appendix IX semi-volatile organics results (produced for the Area 36 soil sample) indicate that semi-volatiles are either absent in the Site soils or present only in low concentrations. These results support the application of solidification/stabilization treatment since semi-volatiles can be difficult to stabilize/solidify and may inhibit curing of the treated material.

None of the TCLP results exceeded the RCRA D004 regulatory threshold of 5 mg/l and no other constituents exceed TCLP threshold levels. These results indicate that if solidification/stabilization were to be applied to the Site soils, the TCLP arsenic leachability criterion would be readily met. The solidification/stabilization screening performed by CGEST further supports this conclusion. Here, several conventional binding mixes were tested on soil fines (-200 mesh) from Area 36 and yielded TCLP results well below the 5 mg/l RCRA LDR treatment standard on the stabilized soils (measured levels were less than the method detection limit, 0.1 mg/l). It is important to note that the Area 36 soils analyzed in the CGEST study were also impacted with lead. Typically, lead stabilization requires alkaline conditions, while arsenic stabilization is best performed under more neutral to slightly acidic conditions. For the binders tested, the study showed that a slightly alkaline "balance" can be struck (end point pH's in the range 5.2 to 8.2 under TCLP testing) that will stabilize lead and also will not leach excessive arsenic. This is an important finding, warranting further consideration in the event that fines generated from a soil washing system campaigning a mixture of lead- and arsenic-impacted soils require further solidification/stabilization treatment.

The moisture content results for the Site soils were fairly variable, ranging from approximately 5% to 10% for Area 18 (by inference from results for the lead characterization study sample from the same Area) and Area 36. Moisture content is the most sensitive input variable in solidification/stabilization system design and requires careful control during implementation. At the levels measured in this case, based on the CGEST solidification/stabilization screening work where moisture contents were in the range of 40% to 50%, a requirement for make-up water is indicated.

### **PHYSICAL SCREENING/SOIL WASHING**

The characterization test results of most relevance for the evaluation of physical screening/soil washing remediation technologies are silt/clay content, arsenic distribution with regard to particle size, surface area calculations, Atterberg Limits, organic carbon, GANC/RGANC, MANC/RMANC, and Sequential Extraction.

The silt/clay content of the Site soils, ranging between eight and ten percent for the samples tested, is well below the 40% limited generally considered likely to limit the effectiveness of soil washing processes. The arsenic content in the silt/clay fraction of the soils is substantial at between 51% to 71% (corresponding concentrations range between 186 and 2,550 mg/kg) and suggests that significant beneficial volume reduction of arsenic-impacted soil could be achieved by separating the fine material from the whole soil.

Particle size distribution test results and corresponding chemical results for separated size fractions under the Hazen and Hart Crowser wet screening studies indicate that between approximately 79% and 92% of the soil arsenic is concentrated in the sand and silt/clay fractions. These fractions represent between 27% and 56% of the whole soil. These results indicate that recovery of the cobbles and gravel fraction (representing between 44% and 73% of the whole soil) as "clean" material could be readily achieved using conventional wet screening or classification-type soil washing.

For water-based soil washing processes, the wet screening and Sequential Extraction test results indicate that, due to the low aqueous solubility of arsenic in Site soils under near-neutral pH conditions, little or no water treatment provisions would be necessary.

The surface area calculations for each of the soil size fractions of the Area 36 sample show, as expected, that the majority of the total soil surface area is contained in the silt/clay (~80%), with less in the sands (~20%), and least in the cobbles/gravel (<2%). The arsenic distribution by size fraction for the Area 36 sample increases with reducing size fraction and increasing surface area; these results indicate that arsenic bonding and binding is mostly surface related.

The Atterberg Limits results for the Area 36 soil sample show (as seen generally for all other Site samples tested) that the "sand" soil is non-plastic and should be amenable to processing by physical screening equipment and easy to handle by conventional earth-moving equipment.

The organic carbon content of the Area 36 soils is 0.9%, significantly below the 10% limit generally considered as the value above which complications in a soil Washington system can arise due to contaminant/carbon bonding. Although organic carbon data for the other arsenic characterization soil samples are not available, information from the lead characterization study (Section 5.3) indicates that the organic content of soils across the Site does not vary significantly. The inclusion of an organic matter removal unit operation in a soil washing process to address soil organic carbon should not be necessary.

The GANC/RGANC results show that the Site soils have little buffering capacity and will not require the addition of large amounts of base in a soil-washing scenario to achieve a desired pH change.

In the consideration of MANC/RMANC equilibrium arsenic concentrations, high acetic acid concentrations (approximately 14 equivalents of acid/kg soil) were required to leach arsenic, whereas RGANC indicated that about two base equivalents/kg soil were required for optimum leaching. One stage acidic/alkaline leaching at 20:1 liquids/solids ratio can remove two to four percent of the arsenic using 2N acetic acid, but amounts of between 74% and 80% can be removed using 2N sodium hydroxide. These results indicate that chemical leaching using alkaline reagents has potential for use in a soil washing process to clean the Site soils, possibly in conjunction with wet screening and other physical treatment techniques. Conversely, mildly acidic conditions might be employed in a soil washing treatment scenario to ensure that arsenic remains concentrated in the fines fraction, which could subsequently be stabilized.

Sequential Extraction data show that arsenic is predominantly present, in the sized fractions examined, in association with iron and manganese oxides and organic material. The results indicate that arsenic



species are not soluble in water or under acidic conditions, but can be solubilized under caustic conditions (the organic matter sequential extraction step uses sodium hydroxide and phosphate as the leachant). This speciation information supports the conclusion that chemically enhanced (basic) soil washing or leaching treatment could be used to liberate arsenic from the soils.

The results of the Weyerhaeuser ATS leaching study corroborate the leaching data generated under the Hazen study, and further demonstrate the potential for a chemically enhanced soil washing application, in that they show alkaline solutions to be effective in removing arsenic from the Site soils. In single-stage batch extractions, solutions of sodium hydroxide at approximately pH 11.5 removed up to 57% of arsenic from sand fractions containing starting concentrations of around 100 mg/kg; sodium carbonate solutions gave similar results but required significantly higher reagent doses. A strong dependency on pH was shown in the leaching experiments and there was no evidence that leaching performance was constrained by arsenic solubility. A batch extraction examining relative leaching efficiencies between sub-fractions of the tested soil showed a somewhat greater performance for the larger sub-fraction, and no significant improvements in overall leaching performance were seen in simulated multi-stage countercurrent tests. These various issues will require further investigation before decisions can be made for including leaching in a soil washing treatment application. Similarly, issues including solids hold-up times, temperature and aqueous extract treatment/recycling will need to be addressed.

### **SMELTING/THERMAL RECOVERY**

The characterization test results of chief importance for the evaluation of smelting/thermal recovery remediation technology are total arsenic concentrations, moisture content, RCRA F039 listed metals, and soil mineralogy results.

Cost effective treatment of RCRA D004 waste by secondary smelters requires that arsenic content in the feed soil be on the order of that for lead recovery, i.e., 6-8% by weight. Since total average arsenic concentrations in the Site soils lie in a range of only 29 to 353 mg/kg (i.e., 0.035% maximum), and even the highest measured concentration in the silt/clay fraction is only 0.25%, the soils would not be suitable for smelting without intensive pre-processing to concentrate arsenic into the finer soil fractions. Because of the relatively low arsenic concentrations, the likelihood that smelting would be suitable to treat concentrated soil washing residuals is low.

The maximum acceptable moisture content of a smelter feed soil is 10%. This may place a further constraint on the smelting application for the Site soils; concentrated fines/residuals from a soil washing process would probably need to be passed through a dewatering step.

The RCRA F039 listed metals analyses indicate the presence of antimony, barium, chromium, copper, nickel, selenium, vanadium, zinc, and lead. Though present at low concentrations, these metals are potential impurities, which could adversely impact smelting/thermal recovery processes.

The XRF analysis indicates the presence of a silica-alumina-iron oxide matrix with minor amounts of sodium, magnesium, potassium, phosphorus, sulfur, titanium, and calcium. The presence of these elements is expected in sandy and silty soils. They could, however, adversely affect the viability of smelting/thermal recovery as impurities in the system.



**Table C-1 – Soil Characterization Procedures by Category**

<b>Category</b>	<b>Test Group</b>	<b>Information Use</b>	<b>Test Types</b>
Whole Soil	PHYSICAL HANDLING	Provides information on how soil will behave during excavation and in treatment processes.	Surface Area Calculations (Lead only) Particle Size Distribution Soil Plasticity (Atterberg Limits) USDA Soil Classification Density (loose and packed) Moisture Content
	REGULATORY	Provides information to enable regulatory waste code classification.	Total Constituent Concentration TCLP Constituent Concentrations RCRA Appendix IX Total Metal Concentration TCLP (inorganics and semiVOCs) RCRA Appendix IX Inorganics RCRA Appendix IX SemiVOCs pH
	SOIL CHEMISTRY	Provides information on contaminant/soil interactions to further general understanding of soil properties and to be used in evaluating treatment technologies.	Bonding/Binding Anions Soluble Cations Carbon (total/inorganic/organic) GANC/RGANC

**Table C-1 – Soil Characterization Procedures by Category (Continued)**

Category	Test Group	Information Use	Test Types
Whole Soil (Continued)	SOIL CHEMISTRY (Continued) -Anions	Provides an indication of availability of bonding/binding sites for heavy metals (anions can combine with metals to form low solubility salts).	Sulfide Sulfate Phosphate Carbonates/Bicarbonates
	-Soluble Cations	Can compete with heavy metals for bonding/binding sites (ion-exchange sites, anions).	Calcium Sodium Magnesium Potassium Manganese Iron
	-Carbon Balance	Organic carbon can bond/bind with metals.	Total Carbon Inorganic (fixed) Carbon (by TGA) Organic Carbon (by difference)
	-Alkalinity/Acidity, Buffering Capacity	Provides, and indications of, how soil will respond to acid/base changes.	pH Redox GANC/RGANC

**Table C-1 – Soil Characterization Procedures by Category (Continued)**

<b>Category</b>	<b>Test Group</b>	<b>Information Use</b>	<b>Test Types</b>
Whole Soil (continued)	MOBILITY AND LEACHABILITY	Provides information on mobility in, and availability to, the environment.	TCLP (inorganics and semiVOCs) Equilibrium Leaching Procedure (Lead only) (SAR-F039 List Metals) MANC during GANC/RGANC GANC/RGANC
Soil Fractions	REGULATORY	Provides information on metals of regulatory concern to enable proper management/disposal.	F039 List Metals
	SOIL CHEMISTRY	Provides information on contamination/soil interactions to further general understanding of soil properties and to be used in evaluating treatment technologies.	Cation Exchange Capacity (CEC) (Lead only) Total Iron and Manganese Total Chloride
	SOIL CHEMISTRY -Speciation	Provides information on what specific chemical forms of metals are present and how metals are bound within soil. Aids in determination of impact to environment and technology selection.	Sequential Extraction Scanning Electron Microscopy (SEM) X-Ray Diffraction (SRD) X-Ray Fluorescence (SRF) - Mineralogy



**Table C-2 – Soil Characterization Procedures by Treatment Technology**

<b>Category</b>	<b>Treatment Technology</b>	<b>Test Types</b>
Whole Soil and Fractions	Stabilization/Solidification	GANC/RGANC MANC Total Carbon, Inorganic and Organic Carbon RCRA Appendix IX SemiVOCs TCLP (inorganic and semiVOCs) Moisture Content Speciation Analysis of Fractions
	Physical Screening/Soil Washing	Constituent Distribution with Particle Size Surface Area Calculations Silt/clay Content MANC during GANC/RGANC Organic Carbon Speciation Analysis of Fractions
	Smelting/Thermal Recovery	Total Constituent Concentration Constituent Distribution with Particle Size XRF Scan (mineralogy) Silica Content (by XRF) Moisture Content Total Carbon





Table C-3 – Lead-Soil Characterization Study-Hazen Research-Summary of Test Results

Whole Soil Procedures	Area 7 (Kettle)	Area 18 (Nitrator)	Area 36 (Lead Melt)	Area 40 (Pulverizer)	Area 40 (Glazemill)	Area 40 (Packhouse)
<b>Sample Verification (Pulverized Head)</b>						
Total Lead, mg/kg (avg. of three)	475	180	870	2,110	660	3,570
TCLP Lead	1.7	0.3	4.6	1.3	1.2	21.4
RCRA Hazardous, D008	no	no	no	no	no	yes
<b>Physical Materials Handling</b>						
Particle Size Distribution (cobbles/gravel, sand, silt/clay), %	56/32/12 non-plastic sand	38/53/9 non-plastic sandy loam	61/32/7 non-plastic sand	43/42/15 non-plastic sand	60/32/8 non-plastic sand	53/37/10 non-plastic sand
Soil Plasticity (Atterberg Limits)	1.3/1.4	1.3/1.5	1.6/1.9	1.1/1.5	1.2/1.6	1.1/1.4
USDA Soil Classification	12.2	9.8	5.0	20.5	11.7	17.0
Density (loose/packed), g/cm <sup>3</sup>	4.4	6.0	6.3	7.3	5.4	5.8
Moisture Content, %						
pH (10% solids slurry)						
<b>Regulatory</b>						
Total Lead, mg/kg (RCRA Appendix IX)	380	260	540	1,700	590	3,000
TCLP Lead, mg/l	2.0	0.3	4.7	1.4	0.9	18
RCRA Appendix IX Inorganics	none of concern	none of concern	none of concern	none of concern	none of concern	none of concern
RCRA Appendix IX Semi-VOCs	none of concern	low level semi-VOCs	low level semi-VOCs	low level semi-VOCs	none of concern	none of concern

**Table C-3 – Lead-Soil Characterization Study-Hazen Research-Summary of Test Results (Continued)**

<b>Whole Soil Procedures</b>	<b>Area 7 (Kettle)</b>	<b>Area 18 (Nitrator)</b>	<b>Area 36 (Lead Melt)</b>	<b>Area 40 (Pulverizer)</b>	<b>Area 40 (Glazemill)</b>	<b>Area 40 (Packhouse)</b>
<b>Soil Chemistry</b>						
Bonding/Binding Anions, ppm total	2,600	2,500	2,640	10,650	4,175	7,190
Soluble Cations, ppm total	81	24	77	261	66	94
Carbon (total/inorganic/organic), %	3.3/0/3.3	2.5/0.7/1.8	1.1/0.2/0.9	6.9/0/6.9	4.2/0/4.2	4.2/0/4.2
Net Acid/Base Potential, tons of CaCO <sub>3</sub> /1000 tons of soil	-10.9	-7.5	1.0	2.8	-24.4	-16.1
GANC/RGANC	unbuffered	unbuffered	unbuffered	unbuffered	unbuffered	unbuffered
Equivalents acid to reach pH,4	2	2	2	4	2	2
Equivalents base to reach pH>10	2	2	2	2	2	2
<b>Leaching and Mobility</b>						
TCLP Lead, mg/l	2.0	0.3	4.7	1.4	0.9	18.0
Equilibrium Leaching Procedure						
Leachate lead, mg/l	0.019	0.05	0.032	0.02	0.059	0.064
Equilibrium partition coefficient, K	2.2E+04	1.0E+04	2.0E+04	8.1E+04	1.1E+04	6.3E+04
MANC/RMANC equilibrium lead conc. (acid/base), mg/l	15/7	4/3	33/8	48/14	15/13	90/25

Table C-3 – Lead-Soil Characterization Study-Hazen Research-Summary of Test Results (Continued)

Whole Soil Procedures	Area 7 (Kettle)	Area 18 (Nitrator)	Area 36 (Lead Melt)	Area 40 (Pulverizer)	Area 40 (Glazemill)	Area 40 (Packhouse)
<b>Speciation</b>						
Sequential Extraction	Majority of lead in carbonate, specifically adsorbed, and exchangeable forms	Majority of lead in carbonate, specifically adsorbed, and exchangeable forms	Majority of lead in carbonate, specifically adsorbed, and exchangeable forms	Majority of lead associated with carbonates, iron and manganese oxides, and organic material	Majority of lead associated with carbonates, iron oxides, and organic materials	Majority of lead associated with carbonates, iron and manganese oxides, and organic material
SEM/XSD	Positive ID of lead sulfate and lead sulfide	Positive ID of lead carbonate, lead oxide, and lead-manganese oxide	Positive ID of lead carbonate, lead silicate, metallic lead, and lead-iron oxide	Positive ID of lead carbonate, lead silicate, lead oxides, and lead-iron oxide	Positive ID of lead silicate, lead oxides, lead phosphate, and lead-iron oxide	Positive ID of lead oxides (manganese, tin, aluminum, and iron)
<b>Soil Chemistry</b>						
Cation Exchange Capacity, Meq/100g Chloride, mg/kg Iron/Manganese, mg/kg	34 100 23,350/620	84.2 <100 34,238/838	49.6 <100 39,979/775	98.1 500 63.630/990	111.0 100 30,050/1,150	95.9 260 95,820/1,090
<b>Regulatory</b>						
F039-List Metals	As, Pb, Ba, Cr	As, Pg, Ba, Cr	As, Pb, Ba, Cr	Pb, Ba, Cr, Zn	As, Pb, Zn	As, Pb, Ba, Cu, Zn

**Table C-3 – Lead-Soil Characterization Study-Hazen Research-Summary of Test Results (Continued)**

<b>Whole Soil &amp; Fraction Procedures</b>	<b>Area 7 (Kettle)</b>	<b>Area 18 (Nitrator)</b>	<b>Area 36 (Lead Melt)</b>	<b>Area 40 (Pulverizer)</b>	<b>Area 40 (Glazemill)</b>	<b>Area 40 (Packhouse)</b>
<b>Physical Screening/Soil Washing</b>						
Silt/Clay Content, %	12	9	7	15	8	10
Lead Distribution by Particle Size (% of total lead in cobbles/gravel, sand, silt/clay)	16/29/55	3/30/67	10/63/27	5/56/39	13/48/39	3/58/39
Total Lead Conc. By Particle Size (cobbles/gravel, sand, silt/clay), mg/kg	130/420/2,170	50/310/3,970	180/2,100/3,980	195/2,310/4,350	130/950/3,320	130/4,260/10,600
Surface Area Calculations (% of total surface area in cobbles/gravel, sand, silt/clay)	<1/17/83	<1/36/64	2/22/76	<1/18/82	1/23/76	1/20/79
Organic carbon, %	3.3	1.8	0.9	6.9	4.2	4.2
GANC/RGANC Equivalents acid to reach pH<4 Equivalents base to reach pH>10	Unbuffered 2 2	Unbuffered 2 2	Unbuffered 2 2	Unbuffered 4 2	Unbuffered 2 2	Unbuffered 2 2
MANC/RMANC equilib. lead Conc. (acid/base), mg/l	15/7	4/3	33/8	48/14	15/13	90/25
% Lead Leached under MANC/RMANC (acid/base)	60/29	44/30	75/18	45/13	45/40	50/14

Table C-3 – Lead-Soil Characterization Study-Hazen Research-Summary of Test Results (Continued)

Whole Soil & Fraction Procedures	Area 7 (Kettle)	Area 18 (Nitrator)	Area 36 (Lead Melt)	Area 40 (Pulverizer)	Area 40 (Glazemill)	Area 40 (Packhouse)
<b>Stabilization/Solidification</b>						
GANC/RGANC MANC/RMANC Minimum Lead Concentration (mg/l)pH Range Carbon (total/inorganic/organic), % RCRA Appendix IX SemiVOCs	Unbuffered <1/5.5 to 6.8  3.3/0/3.3 none of concern	Unbuffered <1/4.6 to 6.1  2.5/0.7/1.8 low level semiVOCs	Unbuffered <1/4.8 to 8.5  1.1/0.2/0.9 low level semiVOCs	Unbuffered 1/6.3 to 12.5  6.9/0/6.9 low level semiVOCs	Unbuffered 1/5.1 to 8.9  4.2/0/4.2 none of concern	Unbuffered 1/6.3 to 7.8  4.2/0/4.2 none of concern
TCLP Lead, mg/l Moisture Content, %	2.0 12.2	0.3 9.8	4.7 5.0	1.4 20.5	0.9 11.7	18 17.0
<b>Smelting/Thermal Recovery</b>						
Total Lead, mg/kg Lead Distribution by Particle Size (% of total lead in cobbles/gravel, sand, silt/clay) Total Lead Conc. By Particle Size (cobbles/gravel, sand, silt/clay), mg/kg Silica Content (weight % in Cobbles/gravel, sand, silt/clay) Moisture Content, % Total Carbon, %	380 16/29/55  130/420/2,170  -/69/59 12.2 3.3	260 3/30/67  50/310/3,970  -/-/33 9.8 2.5	540 10/63/27  180/2,100/3,980  -/-/45 5.0 1.1	1,700 5/56/39  195/2,310/4,350  -/56/28 20.5 6.9	590 13/48/39  130/950/3,320  -/-/28 11.7 4.2	3,000 3/58/39  130/4,260/10,600  -/50/29 17.0 4.2



**Table C-4 – Arsenic-Soil Characterization Study-Hazen Research-Summary of Test Results**

<b>Whole Soil Procedures</b>			
<b>Physical Materials Handling</b>	<b>Area 18</b>	<b>Area 36</b>	<b>RR Track Area</b>
Particle Size Distribution (cobbles/gravel, sand, silt/clay), %	66/26/8	60/30/10	52/39/9
Arsenic Distribution by Particle Size (% of total arsenic in cobbles/gravel, sand, silt/clay, slimes)	12/28/35/25	18/30/47/5	7/22/63/8
pH (10% solids slurry)		6.2	
<b>Regulatory</b>			
TCLP Arsenic, mg/l (conducted at 4.9 pH) RCRA Hazardous, D004	<0.03 no	<0.07 no	<0.03 no
<b>Soil Chemistry</b>			
Total Arsenic, mg/kg	41	29	353
GANC/RGANC	unbuffered	unbuffered	unbuffered
Equivalents acid to reach pH<4	>14	>14	2
Equivalents base to reach pH>10	2	<2	2
<b>Leaching and Mobility</b>			
TCLP Arsenic, mg/l	<0.03	<0.07	0.03
MANC/RMANC minimum arsenic Concentration (acid/base), mg/l: pH	0.007:3.8/ 0.05:5.0	0.007:3.7/ 0.035:8.8	0.045:3.2/ 0.28:5.2
MANC/RMANC equilibrium arsenic Concentration (acid/base), mg/l	0.25/2.7	0.06/1.5	2/22
% Arsenic Leached under GANC/RGANC (acid/base)	3/75	4/80	2/74

**Table C-4 – Arsenic-Soil Characterization Study-Hazen Research-Summary of Test Results (Continued)**

<b>Soil Fraction Procedures</b>			
<b>Speciation</b>	<b>Area 18</b>	<b>Area 36</b>	<b>RR Track Area</b>
Sequential	Majority of arsenic associated with the organic fraction and, to a lesser extent, with the non-crystalline iron oxide fractions.	Majority of arsenic associated with the organic and non-crystalline iron oxide fractions and to a lesser extent, with the crystalline iron oxide fraction.	Majority of arsenic associated with the crystalline iron oxide and organic fractions and, to a lesser extent, with the non-crystalline iron oxide fraction.
SEM	Very little arsenic detected, but associated mainly with iron oxide. Also associated with silicates, and present as iron-arsenic-phosphate.	Arsenic associated mainly with iron oxide (including suspected goethite); arsenic also detected in particles (containing Fe, Al, Ca, & Si) attached to carbon-charcoal particles and with lead-manganese-oxide.	Arsenic associated mainly with iron oxide and organic material. Also positive identification of iron-arsenic-phosphate, lead-arsenic-sulfate and potassium-iron-sulfate.
XRF	Presence of silica-alumina-iron matrix	Presence of silica-alumina matrix	Presence of silica-alumina-iron matrix



Figure C-1 – Flow sheet for Sample Preparation and Testing

